Experimental report

17/09/2024

Proposal: 9-12-690 **Council:** 4/2023

Title: EFFECT OF VARIOUS CARBON FILLERS ON THE PROPERTIES OF SEBS BASED ELECTRO-MECHANICAL COMPOSITES

Research area: Soft condensed matter

This proposal is a new proposal

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Samples: SEBS-rGO
SEBS
SEBS/CNT
SBS/G-NPL

Instrument	Requested days	Allocated days	From	To
IN13	6	6	24/10/2023	30/10/2023
D22	1	1	08/11/2023	09/11/2023

Abstract:

Polymer-based nanocomposites are attracting large attention in recent years both in the scientific and industrial areas. Through the inclusion of fillers and using different processing methods, a wide range of polymer properties (mechanical, electrical, thermal, etc.) can be enhanced. Polymer based piezoresistive materials with wide range of conductive nanofillers arise as a suitable sensor for industrial applications. Different behaviour of macroscopic parameters depending of fillers type and concentration are presented in the literature for the piezoresistive composites. For the moment there is no full understanding of observed results due to the lack of structural and dynamic information at nanoscale for these nanocomposites. Here our objective is to correlate the impact on the macroscopic properties of electro-mechanical composites interesting for industrial applications with the effects of fillers on the structure and atomic mobility. We plan to follow the atomic displacements at the ~ 80 ps scale of the polymer matrix in this kind of nano-composites by EFWS on IN13. We will investigate SEBS with 5%wt of carbonaceous nanofillers with different dimensionality characteristics.

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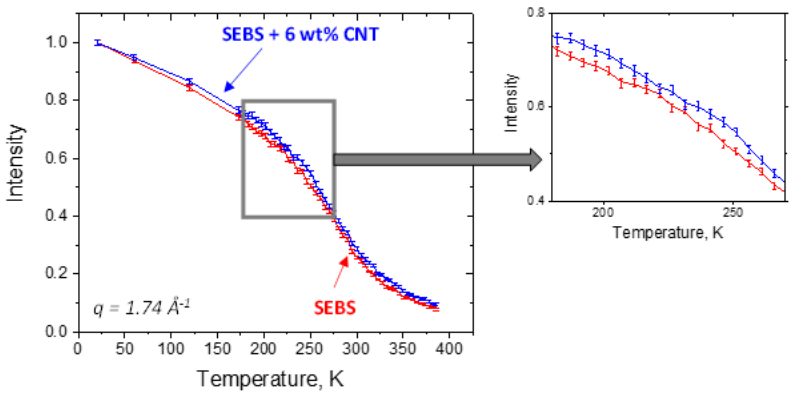
Aim. The aim of this experiment was to investigate the dynamics and nanostructure of electromechanical polymer composites and to correlate the macroscopic properties relevant to industrial applications with the effects of fillers on structure and atomic mobility. The study was conducted using a thermoplastic triblock copolymer, poly(styrene-ethylene-butylene-styrene) (SEBS), with various carbon nanofillers – carbon nanotubes (CNT), graphene nanoplatelets (G-NPL), reduced Graphite Oxide (rGO) and carbon black (CB).

Samples. Nanocomposite films of SEBS with various concentrations of nanofillers were produced using following method. To ensure good dispersion and disintegration of the fillers, they were placed in an Erlenmeyer flask with toluene and ultrasonicated for 30 min. SEBS was then added to the solution and stirred until fully dissolved. Thin (200–300 µm) and highly flexible composite films were obtained by spreading the solution onto a clean glass substrate and allowing the toluene to evaporate at room temperature. The concentration of CNT was varied from 0 to 6 wt%, G-NPL from 0 to 4 wt%, rGO from 1 to 4 wt%, and CB from 0 to 30 wt%.

Measurements. The mobility of protons in the composites was monitored at different q-values using elastic fixed window scans (EFWS) at IN13 spectrometer while heating from 10 to 350 K. The structure of the composites was studied using small-angle neutron scattering (SANS) on the D22 instrument in the q-range of 0.0015 – 0.3 A^{-1}.

Results. EFWS data for neat SEBS and the composites with different fillers show that, in most cases, the dynamics slow down when fillers are introduced. Indeed, the scattered intensity is higher in the presence of fillers than in their absence (Fig. 1).



This is attributed to the reduced mobility (smaller mean square displacements) of protons in the composite due to spatial restrictions imposed by the filler. The strongest influence is observed for 6 wt% CNT.

Fig. 1. EFWS results as a function of temperature for the neat SEBS matrix and the SEBS composite with 6 wt% CNT.

SANS data were acquired to aid the interpretation of the IN13 scans by providing structural information. SANS profiles of the neat SEBS matrix (Fig. 2) show sharp structural peaks, which arise from the self-assembled structure of the block copolymer. Based on these SANS data and additional SAXS measurements obtained in our lab, the

relative positions of the structural peaks correspond to the cylindrical morphology of the microphase separated domains of polystyrene and poly(ethylene-buthylene).

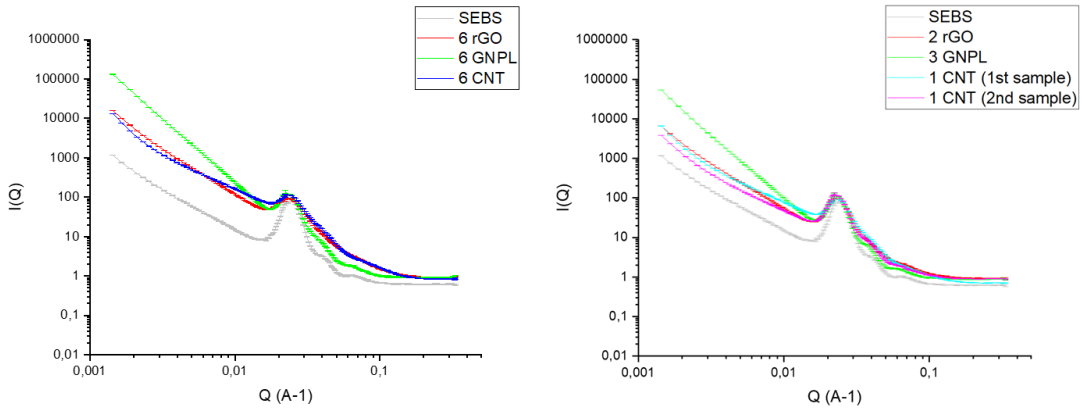


Fig. 2. SANS patterns of the SEBS nanocomposites with various fillers, with the concentrations and types of fillers indicated in the figures.

This is further confirmed by AFM images (Fig. 3). When fillers are added, an increase in intensity is observed in all cases, attributed to the scattering from the fillers. The low-q region of the scattering curves indicates that the fillers are aggregated within the polymer matrix. A detailed analysis of the aggregated structure is currently underway, using combined SANS, SAXS and electron microscopy data. In the presence of fillers, the structural peaks become broader and are obscured by the fillers. However, AFM data (Fig. 3) indicate that the fillers induce changes in the microphase-separated domains. For instance, with 6 wt% CNTs, lamellae are observed instead of cylinders.

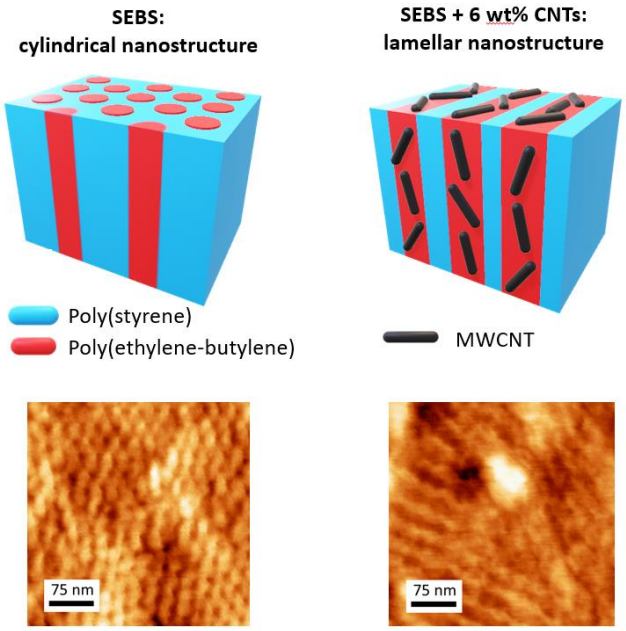


Fig. 3. AFM micrographs of the neat SEBS matrix and the SEBS composite with 6 wt% CNTs, along with a schematic representation of the microphase-separated morphologies.

Conclusion. The dynamics and nanostructure of SEBS composites with various carbon nanofillers were studied using a combined of EFW scans on the IN13 instrument and SANS measurements at D22. The results show that both the dynamics and structure of the composites are altered compared to the neat matrix, due to the interaction between the matrix and the fillers, as well as changes in the microphase-separated morphology of SEBS.